

A RAPID METHOD FOR THE DETERMINATION OF SULPHUR DIOXIDE IN SULFITED POTATO FLAKES

L. R. Ross and R. H. Treadway¹

Abstract

A simple and rapid method is presented for the determination of sulfite in dehydrated potato flakes. Special extraction and filtration techniques are used to prevent potato cell rupture and subsequent release of starch. Presence of cooked starch would interfere greatly with filtration of the extract. An iodimetric titration and simple calculation yield reproducible results with little effort or time.

Introduction

A previous publication by the authors (7) briefly reviews some methods used for determining the sulfur dioxide (SO₂) content of fruit and vegetable products and presents a rapid method for determination of sulfur dioxide in sulfited prepeeled potatoes. By suitable modification of the method developed in this Laboratory for determining the SO₂ content of raw potatoes, it has been found convenient to use the general scheme on potato flakes. Flakes are a form of cooked, dehydrated mashed potatoes developed at the Eastern Regional Research Laboratory (1, 2, 3, 4). This dehydrated potato product is widely used by consumers and in the institutional food service; many millions of pounds of flakes are produced annually. During the processing of potato flakes, sulfite is added to preserve the quality of the dried product. While colorimetric methods employing sophisticated instruments are considered quite satisfactory for determining the SO₂ present in processed foods, some flake processors have expressed the desire for a reliable method employing simple equipment. The method described here is satisfactory for routine use, as in a plant quality control laboratory. The equipment is readily obtained and inexpensive, and the procedure is rapid.

The chief problem in assaying flakes for SO₂ content in comparison with the same determination in fresh potato is that the gelatinized starch in flakes must remain within the cells during the SO₂ extraction. If much of this starch leaves the cells it will produce a high viscosity slurry that is difficult to filter. This has been avoided by controlled mixing of the flakes with the extracting solution and by using cotton cloth (defined below) to remove the fibrous material before filtering through paper.

Materials and Methods

Apparatus:

Magnetic stirrer — variable speed standard size Cole-Parmer No. 1250 "Magnetir" type*, capable of effectively stirring 1 liter of 60% glycerol solution; *Stirring bars* — plastic coated, cylindrical, 2" x 5/16", A. H. Thomas Co. No. 8608 K 25, "Kel-F" coated-type; *Timer* — watch or clock with second hand; *Trip scale or Torsion balance* — capacity 100 g., sensitivity 1/10 g.; *Beakers* — 100, 400, 600, 1000 ml. low form; *Graduated cylinders* — 5, 10,

¹Research Chemist and Assistant Director, respectively, Eastern Regional Research Laboratory, Northeastern Region, Agricultural Research Service, U.S. Department of Agriculture, 600 East Mermaid Lane, Philadelphia, Pennsylvania 19118. Accepted for publication August 28, 1972.

*Mention of specific manufacturers or products does not imply endorsement by the United States Department of Agriculture over others not mentioned.

100, 250, 500 ml.; *Pipettes* — 100 ml.; *Burette* — 50 ml. with 0.1 ml. graduations; *Funnels* — 5 inch diameter; *Funnel rack*; *Filter paper* — folded 300 mm. diameter, Whatman No. 2V type or equivalent; *Dissolving cone* — porcelain, Coors size 1A type, 140 mm. x 140 mm., Macalaster-Bicknell Co. No. 13,576, size 4; *Cotton cloth* — glassware drying towel or equivalent cloth, approx. 60 threads/inch and 5 oz./yd²; U.S. Govt. towels (Int. Fed. Spec. #DDT-T-0050) were used; *Bottle* — 9L, glass-stoppered; dark glass, painted black or stored in dark for iodine solution.

Reagents:

Special buffer solution. Dissolve 35 g. of citric acid monohydrate and 65 g. of disodium phosphate heptahydrate in distilled water. Add 0.5 ml. toluene as a preservative and dilute to a liter with stirring. When diluted to 5 times its volume, as in this determination, this buffer has a pH of 4.4.

Iodine 0.01 N solution. (May be purchased from chemical supply houses.) Dissolve 12 g. of iodine and 22 g. of potassium iodide in a minimum of water with a magnetic stirrer. Dilute to 9 liters and standardize against 0.0100 N sodium thiosulfate solution or against arsenious oxide according to the AOAC method (6). Store the iodine in the dark or in a dark bottle.

Hydrochloric acid. Concentrated ACS grade.

Formaldehyde. 28% solution ACS grade.

1% Starch indicator solution. Mix 10 g. of soluble starch, 25 g. of potassium iodide ACS grade, 10 g. of sodium bicarbonate ACS grade and 1/100 g. of mercuric iodide ACS grade in a minimum of hot distilled water and dilute with the same to a liter with stirring.

Santocel C. (Monsanto Chemical Co.) filter aid.

Procedure:

Stirring speed determination. A standard stirring speed must be obtained. Too low a stirring speed gives incomplete SO₂ extraction while too high a speed breaks potato cells and prevents rapid filtration. With similar rheostat settings, magnetic stirrers of different makes give different stirring speeds. There is even some variation in speed among stirrers of the same make. Variations in the stirring bar size also change the effective speed. A simple procedure can be used to calibrate the rheostat setting for controlling the stirring speed during extractions. Place a 2" x 5/16" magnetic stirrer in a liter low-form beaker, add 700 ml. water and place on the magnetic stirrer. Gradually increase the stirring speed with the rheostat until the vortex tip just touches the stirring bar. When this remains constant for one minute, mark the rheostat setting permanently.

SO₂ Extraction. Place the magnetic stirring bar in a liter low-form beaker, add a 50.0 g. representative sample of the flakes, then 100.0 ml. of the special buffer solution and 500.0 ml. of water. Stir magnetically at the previously determined setting for 10 minutes. Line the dissolving cone with a circular piece of cloth and place it on a liter beaker. Filter the slurry through this and wring out the cloth by hand when the free liquid disappears. Add to the filtrate 100 ml. of Santocel C, measured out in a beaker, and stir the mixture. Filter through the folded filter paper in a funnel into a 400 ml. beaker marked at the 100 ml. level. With slurries yielding first filtrates that are difficult to pass through paper, it is advisable to carry out a second filtration (Santocel added) through cloth. Then the final filtration is done through paper. Pipette a 100.0 ml. aliquot of this filtrate into a 600 ml. beaker, add 200 ml. of water and 5 ml. of 1% starch indicator solution.

Titration. Before attempting this determination, several practice titrations should be carried out in order to become familiar with the unusual but dependable end point. Slow titrations result in undesirable results due to side reactions which must be controlled. The side reactions will be cancelled out if the sample and blank titrations are carried to a similar end point and in a similar manner. The end point for the titration with the 0.01 N iodine solution is the medium blue which just fades out in 20 sec. The titration must be carried out to the end point with the stopcock wide open. Only one or two closely spaced terminal additions should be made, if necessary, and rapid magnetic stirring should be employed.

Blank Determination. To another 100.0 ml. aliquot of the filtrate of the same or similar extraction in a 600 ml. beaker add 2.5 ml. hydrochloric acid, 10 ml. formaldehyde, stir and let stand for 10 min.

Calculations. Subtract the blank titration from the regular titration to get the corrected ml. of iodine solution equivalent to the sulfur dioxide in the aliquot. Calculate the parts per million of SO₂ with this formula:

$$\text{SO}_2 = \frac{\text{corrected ml. iodine} \times \text{normality of iodine} \times .03203 \times 1,000,000}{8.333 \text{ g. sample in aliquot}}$$

$$= 3840 \times \text{corrected ml. iodine} \times \text{normality}$$

Results and Discussion

The procedure described here was used to determine the amount of sulfite present in three experimental samples of potato flakes that had been sulfited during processing. In order to compare the results with those from a recognized method, the same samples were analyzed by the direct colorimetric method of Nury, et al. (5) as modified in this Laboratory for use with dehydrated potato products. Previous experience with this method allowed calculation of a standard deviation of 6.1 ppm SO₂. Values obtained by the titration method were 1670, 516, and 655 ppm SO₂, with standard deviation of 13 ppm for individual repeated determinations which included all steps from sample weighing through titration. Average values for these samples by the colorimetric procedure were 1554, 494, and 689 ppm*. The standard deviation of the average difference between the two methods for the three samples (8) was found to be 55.3. Using the t test, the average difference, 60.7, was found not to be significantly different from zero at the 5% level; t = 1.90, critical value of t, 2 DF, 5% level is 4.30.

Literature Cited

1. Cording, J., Jr., M. J. Willard, Jr., R. K. Eskew, P. W. Edwards, and J. F. Sullivan. 1955. Potato flakes. A new form of dehydrated mashed potatoes. II. Some factors influencing texture. U. S. Dept. of Agriculture, ARS 73-9 (Processed), Agr. Res. Service. Washington, D.C. 20250.
2. Cording, J., Jr., M. J. Willard, Jr., R. K. Eskew, and J. F. Sullivan. 1957. Advances in the dehydration of mashed potatoes by the flakes process. Food Technol. 11: 236-240.
3. Cording, J., Jr., J. F. Sullivan, and R. K. Eskew. 1959. Potato flakes. A new form of dehydrated mashed potatoes. IV. Effects of cooling after precooking. U. S. Dept. Agriculture, ARS 72-25 (Processed), Agr. Res. Service. Washington, D. C. 20250.
4. Eskew, R. K., C. S. Redfield, J. Cording, Jr., M. J. Willard, Jr., J. B. Claffey, P. W. Edwards, and J. F. Sullivan, 1956. Potato flakes. A new form of dehydrated mashed potatoes. III. Estimated commercial cost. U. S. Dept. of Agriculture, ARS 72-12 (Processed), Agr. Res. Service. Washington, D. C. 20250.

*We thank Oksana Panasiuk for these analyses.

5. Nury, F.S., D. H. Taylor, and J. E. Brekke. 1959. Modified direct colorimetric method for the determination of sulfur dioxide in dried fruits. *J. Agr. Food Chem.* 7: 351-353.
6. Official Methods of Analysis of the AOAC. 1970. Association of Official Analytical Chemists, 11th ed., page 874, No. 45.020.
7. Ross, L. R. and R. H. Treadway. 1960. A rapid method for the determination of sulfur dioxide in sulfited pre-peeled potatoes. *Amer. Potato J.*, 37: 102-107.
8. Youden, W. J. 1951. *Statistical Methods for Chemists.* (p. 26). John Wiley & Sons, Inc., New York.